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## (54) Fabric softening compositions containing natural hectorite clay

Textil-Wäscheweichmacher, enthaltend natürlichen Hektoritten

Composition adoucissante textile contenant une argile hectorite naturelle

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(56) References cited:  
EP-A- 0 225 142                    EP-A- 0 297 673  
EP-A- 0 299 575                    GB-A- 1 376 379  
LU-A- 86 451                        US-A- 4 062 647

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**Description****Technical field**

5 The present invention relates to wash or rinse-added fabric-softening compositions. More specifically it relates to such compositions containing a hectorite clay of natural origin, the clay being in the form of particles having a narrowly-defined layer charge distribution.

**Background of the Invention**

10 Smectite clays are known fabric-softening agents, and their use in both detergent compositions and fabric-softening compositions has been disclosed in the art. Representative of this art is GB-B-1 400 898.

15 It is now well recognized in the detergent industry that clays of the type disclosed in GB-B-1 400 898 provide significant fabric softening benefits when used in a laundry detergent. Yet, it is equally well recognized that deposition of these clays onto the fabrics during the laundering process is far from complete; in fact, under typical European laundry conditions, less than half of the available clay is deposited onto the fabrics, the remainder being rinsed away with the laundry liquor during the subsequent rinsing steps. Moreover, the softening effect obtained as a result of the clay deposition is affected by factors that are not well understood.

20 It is an object of the present invention to provide fabric softening compositions, than can be added during the main wash stage, or during the rinse stage of a laundry process, comprising a fabric softening clay from which the clay particles are more efficiently deposited onto fabrics. It is further object of this invention to select clay materials for use in conditioning compositions that provide a significantly better fabric-softening performance than the clay materials used to date in commercial detergent and other fabric softening/conditioning compositions, in particular, which do not necessitate the compulsory presence of an additional softening/conditioning agent.

25 Indeed, in rinse-added fabric softening executions disclosed in the art, smectite clays have usually been used in combination with other ingredients, such as conventional rinse-added fabric softening actives.

Illustrative of this art are :

30 GB-A-1 519 605, disclosing fabric softening compositions containing mixtures of smectite clays and water-insoluble quaternary ammonium compounds;

US-B-4 292,835 describing solid fabric softening compositions containing smectite clay complexed with an anionic surfactant, and fabric softening amines or salts thereof ;

EP-A-0 004 111 describing fabric-care compositions consisting of aqueous dispersion of a smectite-clay and a gelatinized vegetable starch.

35 EP-A 225 142 discloses certain days incorporated into structured liquid compositions which have a defined viscosity which is stable over time.

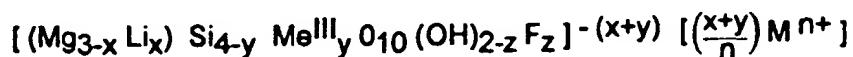
EP-A 297 673 discloses the use of specific hectorite clays in detergent compositions. The compositions also comprise surfactant and builder.

40 Smectite clay agglomerates are also known in the art, such as US-B-4 609 473 and LU-A-86451, disclosing agglomerates of a conventional fabric-softening smectite-clay and sodium sulphate.

45 US-A 4 062 647, issued on December 13th 1977, discloses built granular detergent compositions comprising softening clays to provide fabric softening and anti-static benefits. Clay having particular cation exchange characteristics is selected to provide the benefits. However the problem of effective deposition of clay on to fabric surfaces for softening is not completely solved.

**Summary of the Invention**

50 The present invention relates to fabric-softening compositions containing an hectorite clay of natural origin, said hectorite clay having the general formula



55 where in  $Me^{III}$  is Al, Fe, or B; or  $y = 0$ ;  $M^{n+}$  is a monovalent ( $n=1$ ) or divalent ( $n=2$ ) metal ion, said clay having a layer

charge distribution ( $x+y$ ) such that at least 50% preferably at least 65%, of the layer charge is in the range of from 0.23 to 0.31.

The particulate agglomerates of the invention contain more than 25% and less than 95% preferably at least 50% by weight of the hectorite clay.

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#### Detailed Description of the Invention

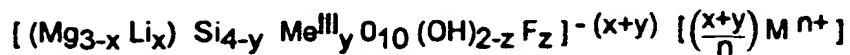
##### The hectorite clay

10 The essential component of the present compositions consists of a particular smectite-type fabric-softening clay. Smectite clays can be described as three-layer expandable materials, consisting of alumino-silicates or magnesium silicates, having an ion-exchange capacity of at least 50 meq/100 g of clay.

15 It has now been found that a specific, narrowly-defined class of smectite clays show an unusually great propensity to deposit onto fabrics, in the rinse step of a washing cycle. The fabric-softening effect derivable from the above-defined hectorite clays is greater than the effect from conventional smectite-type clays or synthetic hectorites.

20 The particular types of smectite clays suitable for the present compositions are hectorites of natural origin, in the form of particles having the general formula

25



25 wherein  $\text{M}^{\text{III}}$  is Al, Fe, or B; or  $y=0$ ;  $\text{M}^{\text{n+}}$  is a monovalent ( $n=1$ ) or divalent ( $n=2$ ) metal ion, for example selected from Na, K, Mg, Ca, Sr.

The present hectorite clays are selected on the basis of their layer charge properties.

30 In the above formula, the value of  $(x+y)$  is the layer charge of the hectorite clay. The hectorite clays suitable for the compositions of the present invention have a layer charge distribution such that at least 50% is in the range of from 0.23 to 0.31.

Preferred are hectorite clays of natural origin having a layer charge distribution such that at least 65% is in the range of from 0.23 to 0.31.

35 The layer charge distribution of the clay material can be determined using its swelling in the presence of cationic surfactants having specific chain lengths. This method is described in detail by Lagaly and Weiss, Zeitschrift fuer Planzenernaehrung und Bodenkunde, 130(1), 1971, pages 9-24.

40 Recently, a method has been developed for objective assessment of fabric softeners. The method consists of a battery of tests, known in the detergent industry as the KES-F system of Kawabata. The method is described in S. Kawabata, "The Standardization and Analysis of Hand Evaluation", 2nd Ed., Textile Mach. Soc. of Japan, Osaka, 1980. Particularly useful to characterize present softening clays is the shear hysteresis parameter 2HG5, one of the parameters of the KES-F system. The shear hysteresis parameter 2HG5 is discussed in more detail in Finnimore and Koenig, Melland Textilberichte 67 (1986) pages 514-516.

45 As noted hereinabove, the clays employed in the compositions of the instant invention contain counterions, such as protons, sodium ions, potassium ions, calcium ions, magnesium ions, and the like. It is customary to distinguish between clays on the basis of one cation predominantly or exclusively absorbed. For example, a sodium clay is one in which the absorbed cation is predominantly sodium.

A calcium clay is one in which the absorbed cation is predominantly calcium.

The hectorite clays of the present invention should preferably be sodium clays, for better softening activity.

50 Sodium clays are either naturally occurring, or are naturally-occurring calcium-clays which have been treated so as to convert them to sodium-clays. If calcium-clays are used in the present compositions, a salt of sodium can be added to the compositions in order to convert the calcium clay to a sodium clay. Preferably, such a salt is sodium carbonate, typically added at levels of up to 5% of the total amount of clay.

Examples of hectorite clays suitable for the present compositions include Bentone EW® and Macaloid®, from NL Chemicals, N.J., U.S.A., and hectorites from Industrial Mineral Ventures.

55 Form of the compositions and conditions of utilization

The compositions of the present invention are particulate agglomerates.

The composition of the invention can be added during the main wash stage or during a rinse cycle of the washing

process.

## COMPOSITIONS IN PARTICULATE FORM

5 Particulate compositions according to the present invention contain more than 25% and less than 95%, preferably at least 50% by weight of the hectorite clay.

The particulate compositions herein are represented by three main executions :

In one execution, the particulate compositions are agglomerates of the clay particles with additional materials. A preferred execution consists of a specific "agglomerate" execution for use especially in a rinse cycle of a washing process, where the clay particles are agglomerated together with a binding/dispersing agent.

10 These agglomerates should have a median diameter of from 75 microns to 2000 microns, preferably a median diameter of from 100 microns and 1250 microns, most preferably from 300 microns to 1000 microns. The clay agglomerates are preferably screened so as to separate agglomerates less than 75 microns, preferably less than 100 microns, and greater than 2000 microns, preferably greater than 1250 microns.

15 Agglomeration methods and equipment suitable for use herein include those methods known in the art. Non-limiting examples of the equipment suitable for agglomeration of clay from smaller particles include a Dravo pan agglomerator, Loedige agglomeration KG/Schugi Blender-Granulator, whirling knife continuous vertical fluidized bed agglomerator, Niro Fluidized Bed agglomerator, Obrian Mixer/Agglomerator, and a Littleford mixer (Littleford Brothers, Inc., Florence, Kentucky, USA, eg. Model FM130D).

20 Other methods and equipment which use larger amounts of water, including the manufacture of agglomerates (as defined herein) directly from a clay slurry, include a spray drying tower, and a prilling tower.

On a laboratory scale, food processors which are widely available to the general public can be used to agglomerate smaller clay particles into agglomerates in the disclosed size ranges.

25 When, according to the preferred execution herein, the particulate compositions consist of agglomerates of clay particles with a binding/dispersing agent, the following methods can be used : an aqueous mixture of water and the binding/dispersing agent can be first prepared and slowly added to the clay while the clay is subjected to the mechanical agitation of the agglomeration equipment.

30 When the clay agglomerates are made from clay slurry, the binding/dispersing agent can be added and mixed with the slurry prior to formation of the clay into relatively small particles by, for example, prilling or spray drying. These particles can then be agglomerated into the agglomerate range defined above. Alternately, agglomerates encompassed by said size range can be formed directly from the slurry by the same general processing methods. In the former case, the particles are preferably agglomerated with the use of an aqueous solution which contains more of a binding/dispersing agent.

35 Preferred binding/dispersing agents are water-soluble inorganic salts. These can include sodium carbonate, sodium sulfate, potassium carbonate, potassium sulfate, magnesium sulfate, lithium sulfate, lithium carbonate, sodium citrate, and sodium sesquicarbonate. Preferred are sodium sulfate and sodium carbonate.

The agglomerates may contain from up to 40%, preferably from 10% to 35% of a water insoluble inorganic salt, based upon the total weight of the agglomerate.

40 Another type of binding/dispersing agent that can be used, alone or in combination with a water insoluble inorganic salt, which can be used at levels of from 0.5 to 70% by weight of the agglomerates includes surfactants. These include surfactants commonly used as detergents in laundry detergents (though they will be present in substantially lower concentrations in the present agglomerates, especially when these are added at the rinse stage). The surfactants suitable for use can comprise an anionic, nonionic, amphoteric or zwitterionic surfactant or a mixture thereof. Nonionic surfactants, or other surfactants, that can interfere with clay deposition should be used in low amounts only, preferably less than 10% of the weight of the agglomerate. Anionic surfactants are preferred. Typical anionic surfactants are the alkyl benzene sulfonates, alkyl- and alkylether sulfates, paraffin sulfonates, olefin sulfonates, alkoxylated (especially ethoxylated) alcohols and alkyl phenols, amine oxides and alpha-sulfonates of fatty acids and of fatty acid esters, which are well-known from the detergency art. In general, such surfactants contain an alkyl group in the C<sub>8</sub>-C<sub>26</sub> range, more generally in the C<sub>8</sub>-C<sub>18</sub> range. The anionic surfactants can be used in the form of their sodium, potassium or triethanolammonium salts : anionic phosphate surfactants are also useful in the present invention. These are surface active materials in which the anionic solubilizing group connecting hydrophobic moieties is an oxy acid of phosphorus. The more common solubilizing groups, of course are -SO<sub>4</sub>H and -SO<sub>3</sub>H. Alkyl phosphate esters such as (R-O)<sub>2</sub>PO<sub>2</sub>H and ROPO<sub>3</sub>H<sub>2</sub> in which R represents an alkyl chain containing from 8 to 20 carbon atoms are useful herein. Suitable nonionic surfactants useful in the present invention include those obtained by the condensation of one to twelve ethylene oxide moieties with a C<sub>10</sub>-C<sub>18</sub> aliphatic alcohol. The alcohol may be completely linear as occurs in petroleum derived alcohols made by oxo-type synthesis. Other nonionic materials are C<sub>14</sub>-C<sub>15</sub> alcohol condensed with an average of s v n ethylene oxide groups. C<sub>12</sub>-C<sub>13</sub> alcohol condensed with an average of about four ethylene oxide groups and then subjected to stripping to remove unethoxylated and low ethoxylated materials, to leave an ethoxylated having a

mean of 4.5 ethylene oxide groups. Suitable zwitterionic materials include derivatives of quaternary ammonium compounds containing an aliphatic straight chain group of 14-18 carbon atoms and a sulfate or sulfonate anionic solubilizing group. Specific examples include 3-N,N-dimethyl-N-hexadecylammonio-2-hydroxypropane-1-sulfonates; 3-(N,N-dimethyl-N-tallowylammonio)-2-hydroxypropane-1-sulfonate; 3-(N,N-dimethyl-N-tetradecylammonio)-propane-1-sulfonate; and 6-(N,N-dimethyl-N-hexadecylammonio)-hexanoate.

#### OPTIONAL ADDITIVE INGREDIENTS

In the present invention, it is often desirable that to the hectorite clay be added one of several of the ingredients described hereinafter.

In the agglomerate executions of the present invention, the following additives will be used either in lieu of the binding/dispersing agents described above, or, if needed, in addition to these binding dispersing agents, in the specific rinse-added execution described above.

#### 15 Clay flocculating agent

Clay-flocculating agents are very well known in industries like oil well drilling, and for ore flotation in metallurgy. Most of these materials are fairly long chain polymers and copolymers derived from such monomers as ethylene oxide, acrylamide, acrylic acid, dimethylamino ethyl metacrylate, vinyl alcohol, vinyl pyrrolidone, ethylene imine. Gums, like 20 guar gum, are suitable as well.

Preferred are polymers of ethylene oxide, acrylamide, or acrylic acid. It has been found that these polymers enhance the deposition of a fabric softening clay if their weights (weight average) are in the range of from 100,000 to 10 million. Preferred are such polymers having a (weight average) molecular weight of from 150,000 to 5 million.

The most preferred polymer is poly(ethylene oxide). Molecular weight distributions can be readily determined using 25 gel permeation chromatography, against standards of poly(ethylene oxide) of narrow molecular weight distributions.

The compositions herein may comprise, from 0.05% to 20%, by weight of the clay, of clay flocculating agent, if its molecular weight is 150,000 - 800,000 and from 0.005% to 2%, by weight of the clay, if its molecular weight is from 500,000 to 5 million.

Clay-flocculating agents can be used in both aqueous dispersion and particulate forms of the present compositions.

#### 30 Other dispersing aid

Other agents suitable as dispersing aids, in both aqueous dispersions and particulate compositions herein, can be advantageously selected from polycarboxylates of relatively low molecular weight (as compared to above clay-flocculating polymers). Such polycarboxylates can be copolymers of an unsaturated polycarboxylic acid such as maleic acid, as first monomer, and an unsaturated monocarboxylic acid such as acrylic acid as second monomer. Preferably, still, the polycarboxylates herein are homopolymers, having a molecular weight in the range of from 1000 to 10,000, most preferably polyacrylates. Such polycarboxylate dispersing aids may be used at levels of from 10% to 100%, 40 preferably 20% to 50% by weight of the clay, in an aqueous dispersion execution, or from 5% to 50% by weight of the total composition in a particulate composition execution.

#### Stabilizing agent

If a clay-flocculating agent is used in the present compositions, the presence of a stabilizing agent will be desirable. 45 Such a stabilizing agent can be selected from conventional metal sequestering and chelating agents, well known used in the detergency art. Preferred for use herein are chelating agents, such as amino phosphonic acids and salts thereof.

Preferred are ethylene diamine tetramethylenephosphonic acid, hexamethylene diaminetetramethylene phosphonic acid, diethylene triaminepentamethylene phosphonic acid, amino-trimethylene phosphonic acid, and salts thereof.

Above stabilizing agent can be used at levels of from 0.1% to 5% by weight of the clay.

#### Organic humectant

An organic humectant may also be used in the compositions of the present invention. 55 Organic humectants may be any of the various water soluble materials utilized for such a purpose. The organic humectants are preferably selected from the group consisting of a) aliphatic hydrocarbon polyols having from 2 to 9 carbon atoms; b) ethanol alcohols derived from the polyols of a); c) ester alcohols derived from the polyols of a); d) mono- and oligosaccharides; and mixtures thereof.

Higly preferred humectants include glycerol, ethylene glycol, propylene glycol and the dimers and trimers of glycerol, of ethylene glycol and of propylene glycol.

The compositions herein may comprise humectant levels from 0.5% to 30%, preferably from 2% to 15%, by weight of the clay.

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#### Other ingredients

Other optional ingredients which can be used in the present compositions include ingredients such as perfumes, dyes, preservatives, germicides, anti-yellowing agents, and also:

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- for the particulate compositions, especially the agglomerates, organic adhesives such as dextrin, gelatine, carboxymethylcellulose, starch.
- for the aqueous dispersions, antisettling agents such as organo-clays, like the Bentone<sup>R</sup> family, or fumed silicas.

15

#### Additional softening ingredients

The textile-softening compositions of the present invention preferably contain only the present hectorite clay as softening agent; alternatively, the present compositions may further contain, in addition to the clay material, an organic softening agent.

20

When the present compositions are used in the rinse stage of a washing process, the co-softening agents can be chosen among well-known fabric-softening materials typically used in rinse-added softening compositions and mixtures thereof. Such materials include quaternary ammonium compounds, such as ditallowdimethylammonium chloride, and similar compounds where at least one of the tallow chain is interrupted by an ester linkage such as described in EP-A-293 952. Also useful as co-softening agents are also the amines disclosed in EP-A-199 383, in particular the substituted cyclic amines disclosed therein. Suitable are imidazolines of the general formula 1-(higher alkyl) amido (lower alkyl)-2-(higher alkyl)imidazoline wherein higher alkyl is alkyl having from 12 to 22 carbon atoms, and lower alkyl is alkyl having from 1 to 4 carbon atoms. A preferred cyclic amine is 1-tallowamidoethyl-2-tallowimidazoline.

When the present compositions are used in the main cycle of a washing process, the following softening-through-the wash organic co-softening agents may be used:

- amines of the formula R<sub>1</sub>R<sub>2</sub>R<sub>3</sub>N, wherein R<sub>1</sub> is C<sub>6</sub> to C<sub>20</sub> hydrocarbyl, R<sub>2</sub> is C<sub>1</sub> to C<sub>20</sub> hydrocarbyl, and R<sub>3</sub> is C<sub>1</sub> to C<sub>10</sub> hydrocarbyl or hydrogen. A preferred amine of this type is ditallowmethylamine.
- amides of the formula R<sub>10</sub>R<sub>11</sub>NCOR<sub>12</sub>, wherein R<sub>10</sub> and R<sub>11</sub> are independently selected from C<sub>1</sub>-C<sub>22</sub> alkyl, alkenyl, hydroxy alkyl, aryl, and alkyl-aryl groups; R<sub>12</sub> is hydrogen, or a C<sub>1</sub>-C<sub>22</sub> alkyl or alkenyl, an aryl or alkyl-aryl group. Preferred examples of these amides are ditallow acetamide and ditallow benzamide.

To avoid negative interactions with the clay materials, above organic co-softening agents, if used, are preferably (releasably) encapsulated by suitable materials which, while ensuring the proper release of the organic material in the wash or rinse water, remain stable and avoid negative interactions, upon storage of the products.

#### INDUSTRIAL APPLICATION

The following examples are intended to illustrate the main executions of the present invention, without intending to be limitative.

#### Agglomerates

##### EXAMPLE I (first execution)

30kg of a natural hectorite clay (\*) are added to a Loedige agglomerating equipment. Deionized water (+/- 5kg) is sprayed onto the clay powder till agglomeration is reached. The wet agglomerates are dried and sieved to a desired particle size (pref. 100% through 1.68 mm (10 mesh) and 0% through 0.149 mm (100 mesh)- Standard Tyler Sieves). The agglomerates are subsequently dyed and perfumed.

(\*) Bentone EW as in Ex I and II.

EXAMPLE II (second execution)

An intimate mixture of 24kg of a natural hectorite clay powder (\*) and 0.6kg of PEO clay-flocculating polymer (\*\*) is added to a Loedige agglomerator. A mixture of deionized water (+/- 4kg), glycerol (0.65kg) and the sodium salt of Ethylenediamine tetramethylene phosphonate (0.6kg of a 25% solution) is sprayed onto the clay/PEO mixture. Extra deionized water is eventually added to obtain suitable agglomeration. The wet agglomerates are dried, sieved, dyed and perfumed.

EXAMPLES III to VIII (third execution)

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The following compositions are preferred

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| Ingredient (all wt. percentages) | Example # |     |     |       |       |
|----------------------------------|-----------|-----|-----|-------|-------|
|                                  | III       | IV  | V   | VI    | VII   |
| Bentone EW® (NL Industries)      | 90%       | 67% | 90% | 76.5% | 76.5% |
| Sodium Carbonate                 | 10%       | 33% | -   | -     | 15.0% |
| Sodium Sulfate                   | -         | -   | 10% | 15.0% | -     |
| Silica/dye composite             | -         | -   | -   | 8.5%  | 8.5%  |

In the formulations above the Bentone EW® can be replaced, in whole or part, with Macoloid® (NL Industries), IMV Hectorite (Industrial Mineral Ventures), while still providing excellent results.

The compositions can be prepared by agglomerating the clay in a commercial food processor, or other agglomeration equipment known in the art, with a solution of the salt dissolved in deionized water (e.g. 15.0 g salt per 30.0 ml water). The salt solution should be slowly added during the agglomeration procedure. The resulting product can be air-dried at ambient temperature.

Optionally, a water-soluble dye can be incorporated into the composition. This can be done, as in Example VI and VII, by stirring a carrier, such as formed silica gel particles (e.g. Syloid® R 234), with the agglomeration equipment and slowly adding a dye solution (e.g. 1.0 gram of F.D. & C Blue #1 per 30 ml of deionized water), at a final dye to silica weight ratio of about 1.0% until the desired dye level (relative to the total weight of the composition) is obtained. The silica/dye particles can then be agglomerated, preferably with an aqueous salt solution (15.0 g Na<sub>2</sub>SO<sub>4</sub> in 30.0 ml deionized water), air dried, and admixed with the clay particles.

The agglomerated clay and silica/dye particles are screened with testing sieves known in the art to, separate agglomerates less than 100 microns and greater than 1250 microns.

In Examples I to VII, the hectorite is used in its sodium form. The clay can also be used in its calcium form and converted to its sodium form during agglomeration, as shown in Ex. VIII.

EXAMPLE VIII

40

20kg of a natural Turkish calcium hectorite clay powder and 0.4kg of sodium carbonate are well mixed prior to their addition to a Loedige agglomerating equipment. +/-4kg of deionized water is used for the agglomeration. The wet agglomerates are dried, sieved, dyed and perfumed.

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Aqueous Dispersions:EXAMPLE IX

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2g of a natural hectorite clay (\*) is added - under vigorous mixing - to 97.5g of deionized water. High speed mixing is maintained till complete dispersion of the clay. Dyestuff and perfume are added, to make up 100%.

When applied in the rinse step of a laundry program, such a fabric softening composition delivers very significant softness benefits.

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(\*) Macoloid® clay ex Hector CA (NL Chemicals) Sodium form

(\*\*) polymer of ethylene oxide / MW = 300,000

(\*) Bentone EW®: a highly purified hectorite clay from Hector CA (USA), available from NL Chemicals, NJ. The clay particles have a lath shape and a length/width ratio of 10:1 or higher (TEM data). Layer Charge Distribution : more than 65% in the range of from 0.23 to 0.31; sodium form.

EXAMPLE X

To 93.13g of deionized water, 2.27g of a low molecular weight polyacrylate (\*) is added under moderate mixing conditions. High speed mixing is then used to optimally disperse 4g of a natural hectorite clay (\*\*). After dispersing, dyestuff and perfume are added (0.6g) to finish the composition.

PERFORMANCE MEASUREMENT

## Relative Deposition Measurement

## A. Washing procedure:

Prewash: Cotton/Polyester (86%/14%) terry cloths (Style 4025, Dundee Mills, Griffin, GA) that are 11 X 11 square inches (27.9 x 27.9 square cm) and weigh about 50g each are used for the Relative Deposition test. The cloths are washed two times with a conventional non-clay containing detergent formulation (shown below) in 0 g/l (0 grain/gallon) water at 125°F (52°C) for 12 minutes each, then washed two times in 0g/l (0 grain/gallon) water at 125°F (52°C) without detergent and dried in a Whirlpool 3 Cycle Portable Dryer (Model #LE4905XM, Whirlpool Corp., Benton Harbor, MI).

| Prewash Detergent Composition: |  |                    |
|--------------------------------|--|--------------------|
|                                | Ingredient   | % (Wt.)            |
|                                | C <sub>12</sub> Linear Alkyl Benzene Sulfonate (Na Salt) | 4.1                |
|                                | Tallow Alcohol Sulfate (Na Salt)                         | 5.0                |
|                                | Neodol <sup>R</sup> 23-6.5 (Alkyl Ethoxylate)            | 2.0                |
|                                | Tallow Soap  | 1.9                |
|                                | Sodium Tripolyphosphate                                  | 32.0               |
|                                | Silicate   | 6.5                |
|                                | Water and Miscellaneous                                  | --balance to 100-- |

Test Wash: A miniwasher with five pots (such as those manufactured by Yorktown Tool & Die Corp., Yorktown, IN) is used. For wash added clay softener tests, 9.12g of detergent product (Testwash Detergent Composition, as shown below) and 0.58g of a clay of the present invention (77ppm in the wash) are added to 7.57l (two gallons) of 0.103 g/l (6 grain/gallon) water at 95°F (35°C) in each mini-washer pot and agitated for two minutes. Alternately, where specifically set forth herein, higher clay concentrations, eg. 150 ppm, can be utilized. This, of course, will affect results and direct comparisons between clay concentrations are not reliable. A load of fabrics weighing about 341g and including test fabrics of four of the prewashed terry cloths, six polyester/cotton (65%/35%) 11 x 11 square inch (27.9 x 27.9 square cm) swatches (product #7435, Test Fabrics, Middlesex, NJ) weighing a total of about 37g, three 11 X 11 inch (27.9 x 27.9 cm) nylon swatches (product #322, Test Fabrics) weighing a total of about 18g, three 11 X 11 inch (27.9 x 27.9 cm) polyester swatches (product #720-H, Test Fabrics) weighing a total of about 44g, and one polyacrylic sock (Burlington Socks, Balfour Inc., Asheboro, NC) weighing about 42g are added to the wash water. The fabrics are washed for 12 min. and spin dried for two minutes. The fabrics are then rinsed with 7.57l (two gallons) of 0.103 g/l (6 grain/gallon) water at 70°F (21°C) for two minutes, spin dried for two minutes, and dried in a Whirlpool 3 Cycle Portable (Model No. LE4905XM, Whirlpool Corp., Benton Harbor, MI). This test wash procedure is repeated for a second cycle, and the Relative Deposition is measured as described below.

| Test Wash Detergent Composition |   |         |
|---------------------------------|---|---------|
|                                 | Ingredient  | % (Wt.) |
|                                 | C <sub>13</sub> Linear Alkyl Benzene Sulfonate                              | 9.0     |
|                                 | C <sub>14-15</sub> Alkyl Sulfate  | 9.0     |
|                                 | Neodol <sup>R</sup> 23-6.5T (Alkyl ethoxylate)<br>(Mfg. by Shell Chem. Co.) | 1.5     |
|                                 | Sodium Tripolyphosphate   | 38.4    |
|                                 | Silicate  | 14.6    |

(\*) Na - polyacrylate : 44% pure/MW = 4500.

(\*\*) Bentone EW® as in EX I.

(continued)

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| Test Wash Detergent Composition |                    |
|---------------------------------|--------------------|
| Ingredient                      | % (Wt.)            |
| Sodium Carbonat                 | 21.3               |
| Water and Miscellaneous         | --balance to 100-- |

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B. Relative Deposition Measurement

The deposition of the clay containing compositions is calculated based on the deposition of silicon (Si) of terry cloth swatches washed with the test wash detergent composition relative to terry cloth swatches that were prewashed but not subjected to the test wash procedure (blank swatches). Silicon deposition is determined by measurement of the X-ray fluorescence of the silicon. Each Silicon fluorescence is measured in the following manner: An EDAX 9500 X-ray fluorescence unit with a rhodium anode X-ray source (Philips Electronics, Inc., Cincinnati, OH) is used. Each terry cloth swatch is analyzed for 100 live seconds. Count rate of Si (on a per second basis) for each sample is measured and recorded.

Relative Deposition of clay is calculated by the following equation:

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$$\text{Relative Deposition} = \frac{\text{STF} - \text{SFB}}{\text{SW}} \times 1000$$

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wherein, STF is the Si count rate of clay-treated terry cloth fabric, SFB is the Si count rate of blank terry cloth fabric and SW is the Si count rate of a clay sample wafer (pressed clay particles of same area of terry cloth fabric). Count rates of Si for the clay sample wafer and clay deposition on fabric are measured as follows:

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(a) Si count rate for clay sample wafer: The X-ray generator is set at 20 KV/500 microamps. About 2g of clay powder is pressed at about 138 N/mm<sup>2</sup> (20,000 psi) into a pellet with a 27.2 tonne (30 ton) hydraulic press (Angstrom, Inc., Chicago, IL). The sample is rotated during the count rate analysis in a vacuum atmosphere (less than 300 millitorr).

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(b) Si count rate for the terry cloth treated with clay: The X-ray generator parameter is set at 15kV/500 microamps. A disk with a 3cm diameter is cut from a terry cloth swatch. The disk is compressed at about 138 N/mm<sup>2</sup> (20,000 psi) to form a flat smooth disk using a 27.2 tonne (30 ton) hydraulic press, then rotated during the count rate analysis in a vacuum atmosphere.

#### Performance test for rinse added aqueous dispersions

Two aqueous dispersions are prepared;

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Composition A, containing 10g of montmorillonite clay (CSM) activated dispersed in 2 litres water.

Composition B, containing 5g of Bentone EW® hectorite, dispersed in 2 litres water.

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Both compositions are compared for softening performance in the rinse-stage of a washing machine. Tests are conducted in a Miele washing machine, using cotton loads (clean or soiled). Softness is measured after 1 cycle.

#### Results are as follows

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|               | PSU (vs. water alone) |
|---------------|-----------------------|
| Composition A | + 2.8                 |
| Composition B | + 3.5                 |

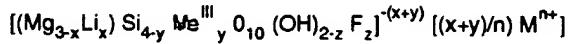
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Above results show that the hectorites of the present invention exhibit a better softening activity, as compared to conventional smectite clay.

**Claims**

1. A wash or rinse-add d fabric soft ning composition in particulate form, containing more than 25% by weight of clay having th g neral formula:

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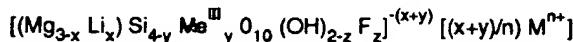
wherein Me<sup>III</sup> is Al, Fe, or B; or y=0; M<sup>n+</sup> is a monovalent (n=1) or divalent (n=2) metal ion, characterised in that the clay is a hectorite clay of natural origin, the clay having a layer charge distribution such that at least 50% of the layer charge is in the range of from 0.23 to 0.31 and wherein the particulate composition is in the form of agglomerates comprising less than 95% by weight of clay, together with additional materials.

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2. A composition according to claim 1, wherein said hectorite clay has a distribution of layer charge (x+y) such that at least 65% of the layer charge is in the range of from 0.23 to 0.31.
3. A particulate composition according to claim 1, wherein the hectorite clay is present at levels of from 50% to 95% by weight.
4. A particulate composition according to claim 3 which is used as a rinse-added composition, and consists of agglomerates, additionally containing from 0.5% to 40% by weight of a binding/dispersing agent.
- 20 5. An agglomerate according to claim 4, wherein said binding/dispersing agent is a water-soluble inorganic salt and said agglomerates comprise from 5% to 40% by weight of said inorganic salt.
6. An agglomerate according to claim 5, wherein said inorganic salt is a sodium salt of a sulfate or a carbonate.
- 30 7. An agglomerate according to claims 4 to 6 wherein said binding/dispersing agent further comprises a surfactant, and said surfactant comprises from 0.5% to 50% by weight of said agglomerates.
8. A composition according to claims 1 to 7, wherein the hectorite clay is in its sodium form.
- 35 9. A composition in accordance with claims 1 to 8 which further contains dispersing aid, preferably a polycarboxylate having a molecular weight from 1000 to 10.000.
10. A composition in accordance with claims 1 to 9 which further contains from 0.005% to 20%, by weight of the total composition, of a clay-flocculating agent.
- 40 11. A composition in accordance to claim 10 wherein the composition further contains an amino-phosphonate chelating agent, at levels of from 0.1% to 5% by weight of the clay.
12. A composition in accordance with claims 1 to 11 which further contains an organic humectant, at levels of from 45 0.5% to 30%, preferably 2% to 15%, by weight of the clay.

**Patentansprüche**

- 50 1. Beim Waschen oder Spülen zuzugebende Textilweichmacherzusammensetzung in Teilchenform, enthaltend mehr als 25 Gew.-% Ton der allgemeinen Formel:



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worin M<sup>III</sup> Al, Fe oder B ist; odery = 0; M<sup>n+</sup> ein einwertiges (n = 1) oder zw iwtiges (n = 2) Metallion ist, dadurch gekennzeichnet, daß d r Ton in Hectoritton natürlichen Ursprungs ist, d rTon eine solch Schichtladungsverteilung aufweist, daß mind st ns 50 % der Schichtladung im Bereich von 0,23 bis 0,31 liegen, und wobei die

teilchenförmig Zusammensetzung in Form von Agglomeraten, die weniger als 95 G. w.-% Ton umfassen, zusammen mit zusätzlichen Materialien vorliegt.

2. Zusammensetzung nach Anspruch 1, wobei der Hectoritton eine solche Verteilung der Schichtladung ( $x + y$ ) aufweist, daß mindestens 65 % der Schichtladung im Bereich von 0,23 bis 0,31 liegen.
3. Teilchenförmige Zusammensetzung nach Anspruch 1, wobei der Hectoritton in Anteilen von 50 bis 95 Gew.-% vorliegt.
- 10 4. Teilchenförmige Zusammensetzung nach Anspruch 3, welche als eine beim Schülen zuzusetzende Zusammensetzung verwendet wird und aus Agglomeraten besteht, die zusätzlich 0,5 bis 40 Gew.-% eines Binde-/Dispergiermittels enthalten.
- 15 5. Agglomerat nach Anspruch 4, wobei das Binde-/Dispergiermittel ein wasserlösliches anorganisches Salz ist und die Agglomerate 5 bis 40 Gew.-% des anorganischen Salzes umfassen.
6. Agglomerat nach Anspruch 5, wobei das anorganische Salz ein Natriumsalz eines Sulfats oder eines Carbonats ist.
- 20 7. Agglomerat nach den Ansprüchen 4 bis 6, wobei das Binde-/Dispergiermittel weiterhin ein Tensid umfaßt und das Tensid 0,5 bis 50 Gew.-% der Agglomerate ausmacht.
8. Zusammensetzung nach den Ansprüchen 1 bis 7, wobei der Hectoritton in seiner Natriumform vorliegt.
- 25 9. Zusammensetzung nach den Ansprüchen 1 bis 8, welche weiterhin ein Dispergierhilfsmittel enthält, vorzugsweise ein Polycarboxylat mit einem Molekulargewicht von 1 000 bis 10 000.
10. Zusammensetzung nach den Ansprüchen 1 bis 9, welche weiterhin 0,005 bis 20 Gew.-% der gesamten Zusammensetzung eines Ton-Flockungsmittels enthält,
- 30 11. Zusammensetzung nach Anspruch 10, wobei die Zusammensetzung weiterhin einen Aminophosphonat-Komplexbildner in Anteilen von 0, bis 5 Gew.-% des Tons enthält.
12. Zusammensetzung nach den Ansprüchen 1 bis 11, welche weiterhin ein organisches Feuchthaltemittel in Anteilen von 0,5 bis 30, vorzugsweise 2 bis 15 Gew.-% des Tons enthält.

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#### Revendications

1. Composition d'assouplissant textile sous forme particulaire, à ajouter au lavage ou au rinçage, ne contenant pas plus de 25% en poids d'argile de formule générale:
- $$[(Mg_{3-x} Li_x) Si_{4-y} Me^{III}_{y} O_{10} (OH)_{2-z} F_z]^{-(x+y)} [(x+y)/n] M^{n+}]$$
- 45 dans laquelle  $Me^{III}$  est Al, Fe ou B; où  $y=0$ ;  $M^{n+}$  est un ion métallique monovalent ( $n=1$ ) ou divalent ( $n=2$ ), caractérisée en ce que l'argile est une argile hectorite d'origine naturelle, l'argile ayant une distribution de charge dans les couches telle qu'au moins 50% de la charge des couches soit dans la gamme de 0,23 à 0,31 et en ce que la composition particulaire est sous forme d'agglomérats comprenant moins de 95% en poids d'argile, avec des matières supplémentaires.
2. Composition selon la revendication 1, dans laquelle ladite argile hectorite a une distribution de la charge dans les couches ( $x+y$ ) telle qu'au moins 65% de la charge des couches soit dans la gamme de 0,23 à 0,31.
  - 55 3. Composition particulaire selon la revendication 1, dans laquelle l'argile hectorite est présente en proportions de 50% à 95% en poids.
  4. Composition particulaire selon la revendication 3, qui est utilisé comme composition à ajouter au rinçage et qui

est constituée d'agglomérés, contenant en outre de 0,5% à 40% en poids d'un agent liant/dispersant.

- 5        5. Aggloméré selon la revendication 4, dans lequel ledit agent liant/dispersant est un sel minéral hydrosoluble et ledit aggloméré comprend de 5% à 40% en poids dudit sel minéral.

- 6        6. Aggloméré selon la revendication 5, dans lequel ledit sel minéral est un sel de sodium d'un sulfate ou d'un carbonate.

- 10      7. Aggloméré selon les revendications 4 à 6, dans lequel ledit agent liant/dispersant comprend aussi un tensioactif, et ledit tensioactif comprend de 0,5% à 50% en poids desdits agglomérés.

8. Composition selon les revendications 1 à 7, dans laquelle l'argile hectorite est sous sa forme sodique.

- 15      9. Composition selon les revendications 1 à 8, qui contient en outre un auxiliaire de dispersion, de préférence un polycarboxylate ayant une masse moléculaire de 1 000 à 10 000.

- 10      10. Composition selon les revendications 1 à 9, qui contient en outre de 0,005% à 20%, en poids de la composition totale, d'un agent de flocculation de l'argile.

- 20      11. Composition selon la revendication 10, dans laquelle la composition contient en outre un agent chélateur aminophosphonate, en proportions de 0,1% à 5% en poids de l'argile.

12. Composition selon les revendications 1 à 11, qui contient en outre un humectant organique, en proportions de 0,5% à 30%, de préférence de 2% à 15%, en poids de l'argile.

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